NASA Technical Memorandum

NASA TM-86540

CONTAINERLESS GLASS FIBER PROCESSING - MSFC CENTER DIRECTOR'S DISCRETIONARY FUND FINAL REPORT

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Science and Engineering Directorate

March 1986

National Aeronautics and Space Administration

George C. Marshall Space Flight Center

TECHNICAL REPORT STANDARD TITLE PAGE

1. REPORT NO.	2. GOVERNMENT ACCESSION NO.	3. RECIPIENT'S CATALOG NO.
NASA TM -86540		

4. TITLE AND SUBTITLE	5. REPORT DATE
Containerless Glass Fiber Processing – MSFC Center Director's Discretionary Fund Final Report	March 1986
	6. PERFORMING ORGANIZATION CODE
7. AUTHOR(S)	8. PERFORMING ORGANIZATION REPORT #
E. C. Ethridge and R. J. Naumann	
9. PERFORMING ORGANIZATION NAME AND ADDRESS	10. WORK UNIT NO.
George C. Marshall Space Flight Center Marshall Space Flight Center, Alabama 35812	11. CONTRACT OR GRANT NO.
	13. TYPE OF REPORT & PERIOD COVERED
12. SPONSORING AGENCY NAME AND ADDRESS	Technical Memorandum
National Aeronautics and Space Administration Washington, D.C. 20546	14. SPONSORING AGENCY CODE

15. SUPPLEMENTARY NOTES

Prepared by Space Science Laboratory, Science and Engineering Directorate.

16. ABSTRACT

An Acoustic Levitation Furnace system is described that was developed for testing the feasibility of containerless fiber pulling experiments. It is possible to levitate very dense materials such as platinum at room temperature. Levitation at elevated temperatures is much more difficult. Samples of dense heavy metal fluoride glass were levitated at 300 °C. It is therefore possible that containerless fiber pulling experiments could be performed. Fiber pulling from the melt at 650 °C is not possible at unit gravity but could be possible at reduced gravities. The Acoustic Levitation Furnace is described, including engineering parameters and processing information. It is illustrated that a shaped reflector greatly increases the levitation force aiding the levitation of more dense materials.

17. KEY WORDS	18. DISTRIBUTION STATEMENT
Acoustic Levitation Fiber Pulling Glass Fibers Fluoride Glass	E. C. Ethridge Unclassified – Unlimited

19. SECURITY CLASSIF. (of this report)	20. SECURITY CLASSIF. (of this page)	21. NO. OF PAGES	22. PRICE
Unclassified	Unclassified	23	NTIS

MSFC - Form 3292 (May 1969)

For sale by National Technical Information Service, Springfield, Virginia 22151

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TECHNICAL MEMORANDUM

CONTAINERLESS GLASS FIBER PROCESSING – MSFC CENTER DIRECTOR'S DISCRETIONARY FUND FINAL REPORT

SUMMARY OBJECTIVES

The objectives of this study were to develop a containerless glass processing system to investigate the feasibility of forming optical communications fibers and/or fiber preforms from containerless melts.

INTRODUCTION

The current signal loss for silica glass optical fibers is 0.2 dB/km in the 1.3 to 1.55 μm wavelength range. This is nearly the practical limit, being very close to the theoretical limit. With silica fibers, one can transmit signals at most 45 to 100 km without repeaters. There is a need for fibers that can transmit much longer distances. An example is for transoceanic cables. If one can eliminate repeaters, then one of the major problems with long cables is solved. Other uses include pyrometry and infrared sensing and mid-infrared optical circuits, such as delay lines, couplers, etc. Long distance applications include non-electrical power transmission, nuclear radiation resistant communications, and ultra-long-length repeaterless data links.

Several years ago theorists predicted that it should be possible for halide compounds or glass fibers to transmit mid-infrared wavelength signals (2 to 11 μm) with a loss of only 0.01 to 0.001 dB/km [4]. Recently, therefore, great interest has been generated for developing these fibers. If the halide optical material can be developed to its theoretical limit as has been done with silica, then cables could be produced in hundreds or thousands of kilometers without repeaters.

A major advantage for containerless processing of glasses is the absence of container-induced contaminants. Glasses melted in crucibles contain trace quantities of their crucible material. These impurities act as absorbing or scattering centers that attenuate the propagation of optical signals and currently are a significant contributor to optical loss in the new optical fiber materials. If one can develop a completely containerless melting and pulling process, it should be possible to totally eliminate crucible contaminants, especially if the glass is never exposed to high temperature prior to the containerless melting. Extension of the process to a low-gravity environment will allow levitation of glasses at much higher temperatures and may allow optical fibers to be pulled at lower viscosities than is possible in Earth's gravity. The latter is believed to be desirable in the processing of certain very low-loss optical fibers.

The Marshall Space Flight Center (MSFC) has supported the development of single-axis interference acoustic levitation furnaces for containerless glass processing in low gravity. Glass fiber and glass preform formation may be a practical application of this type of containerless glass processing technology [5]. It should be noted that optical fibers certainly qualify as a high value, low volume product. A 100-km optical fiber with a diameter of 100 μm has a mass of only a few kilograms.

ULTRA-LOW-LOSS FLUORIDE GLASS

There are four candidate material classes that have potential for ultra-low-loss communication fibers. They include halide glasses, heavy metal oxide glasses, chalcogenide glasses, and crystalline halide compounds. Table 1 illustrates the theoretical and current experimental absorption losses for these materials. The theoretical absorption plotted versus wavelength is illustrated in Figure 1. The fluoride glass fibers have the lowest potential absorption for the amorphous materials while the ultimate lowest loss materials are the crystalline halide salts. Ultra-long fiber production of single crystals has its own unique problems. Intrinsic losses are the ultimate limit for the material while extrinsic losses currently limit the use of the material. Extrinsic losses can conceivably be eliminated.

Optical attenuation consists of absorption and scattering, which are classified into intrinsic and extrinsic factors. Intrinsic optical absorption, α, in dielectric solids occurs by three mechanisms according to equation (1).

$$\alpha = \frac{A}{\lambda^4} + Be^{b/\lambda} + Ce^{-c/\lambda} \qquad (1)$$

where A, B, b, C, and c are constants and λ is the wavelength. The first term represents Rayleigh scattering loss that is caused by microfluctuations in the refractive index of the material. Tran et al. [6] recently demonstrated experimentally that this relationship also applies to the fluoride glasses. The second term is due to ultraviolet absorption originating from electronic bandgap transitions. Both ultraviolet absorption and Rayleigh scattering decrease with wavelength. Infrared absorption, represented by the third term which increases with wavelength, is caused by the coupling of electromagnetic radiation to phonon modes resulting in multiphonon absorption [7]. These individual mechanisms are represented in Figure 2. All of intrinsic absorptions have associated tails; the crossover minimum of the tails ultimately limits the ultimate losses. The total intrinsic spectral loss of a fiber is represented by the summation of the three absorption mechanisms.

Extrinsic absorption losses result from impurities, such as transition-Earth metals, rare-Earth metals, and hydroxyl ions. Material processing resulting in fiber imperfections such as microcrystals, bubbles, stria, and fluctuations in the core-cladding boundary cause extrinsic scattering. These losses are also represented in Figure 1 redrawn from Miyashita and Manabe [7]. Extrinsic losses currently limit the use of the materials, but many of the extrinsic losses can conceivably be eliminted.

To achieve the ultra-low-loss requires ultra-high-purity materials as well as near perfect processing conditions. The levels of transition-Earth and rare-Earth elements must be in the parts per billion level. Likewise oxides must be essentially eliminated. These impurity levels are comparable to that with the existing silica material.

American laboratories at the Naval Research Laboratory and Rome Air Development Center have concentrated on the heavy metal fluorozirconate (ZrF_4) and fluorohafnate glasses (HfF_4). General properties of the heavy metal fluorides such as

zirconium and hafnium fluoride are that the glass transition temperature is generally in the range of 300 to 350 °C with the temperature of maximum crystallization on reheating (T_x) in the range of 400 to 450 °C. Densities are around 4.5 to 5.0 g/cm^3 for zirconium fluoride and 5.5 to 6.0 g/cm^3 for hafnium fluoride. Thermal expansion values are 1.8 to 2 x 10^{-5} [8]. A typical glass composition is represented in Table 1 along with selected physical properties of the ZBLAL fluoride glass. A measure of the glass working range is the difference between the temperature of maximum crystallization and the glass formation temperature ($\Delta T = T_x - T_g$) [9].

Although a recent theoretical study of the feasibility of pulling glass fibers in low gravity [10] concluded that from a heat and fluid flow standpoint there are no advantages to pulling fibers in low gravity. Containerless processing to preserve purity is still a justifiable aim of processing in space. Current and all proposed halide fiber pulling processes utilize crucibles during at least one step of the process. Containerless processing in low gravity allows one to eliminate one or more of the steps requiring the use of crucibles, therefore, reducing the chance of crucible contamination of the melt. Low-gravity containerless processing could limit the crucible contamination inherent in terrestrial techniques.

The glass used in the trial fiber pulling experiments in this study are of the composition given in Table 2 and were supplied by D. Tran et al. [11]. The glasses were melted in a sealed Pt crucible under Ar atmosphere at 800 °C, then transferred in the crucible to anneal at the glass transition temperature.

DESCRIPTION OF THE ACOUSTIC CONTAINERLESS LEVITATION FURNACE

The approach for developing a process and apparatus for studying the formation of glass fibers from containerless melts is to acoustically levitate the molten glass and either consolidate a glass preform or initiate fiber pulling from melt.

A hot wall acoustic levitation furnace was constructed by Intersonics, Inc. of Northbrook, IL. A functional electronics schematic diagram is shown in Figure 3. An acoustic oscillator with a manual frequency control generates a sinusoidal signal with a frequency of approximately 23 kHz. The oscillator has an automatic frequency control circuit which uses a signal from the acoustic driver to adjust the frequency to maximize acoustic transmission from the piezoelectric sound source through the acoustic transformer to the acoustic reflector. The signal from the oscillator is amplified by an acoustic amplifier with a manual power level control. The output is fed to a piezoelectric driver situated below the furnace. The voltage and current of the output signal is examined with an oscilloscope and a power meter.

A schematic representation of the acoustic levitation furnace is shown in Figure 4. The acoustic sound source and driver assembly is positioned below the hot wall box furnace. A piezoelectric sound source is sandwiched between fractional wavelength aluminum resonators. Attached to the resonator is an acoustic transformer that concentrates the acoustic energy and a Ti-6Al-4V alloy acoustic driver. In the top of the furnace is an acoustic reflector that reflects some of the acoustic energy creating interference nodes at which levitation is to occur. The position of the reflector is adjustable with a servomotor assembly to permit real time adjustment of the driver to reflector distance to optimize the levitation force. The reflector

assembly is hollow to permit fiber pulling and quenching through the reflector. Through the side of the furnace is a sample inserter consisting of a steel mesh basket on a retractable arm. This can be used to insert samples into the furnace at temperature without opening the furnace.

The entire acoustic containerless furnace assembly can be contained in a specially constructed dry gas box for experimentation with molten fluoride melts.

STRIP HEATER FIBER PULLING EXPERIMENTS

In order to determine certain general fiber pulling parameters, a strip heater assembly was constructed. The strip heater consists of a step down transformer arrangement to provide the low voltage high current required to heat a platinum foil by joule heating to the desired temperature. The power is applied to water cooled power leads to which a platinum foil strip is connected as in Figure 5. A sample of glass supplied by the Naval Research Laboratory is placed onto the platinum foil and power applied. A variable transformer is used to regulate the power to the furnace to achieve the desired melt temperature, as indicated by a thermocouple in the melt. Fiber pulling is achieved by inserting a platinum wire into the melt and retracting the wire upward.

The strip heater is situated inside a dry glovebox to prevent atmospheric water vapor from reacting with the molten fluoride material. A video camera and recorder system is used to record the fiber pulling experiments.

In general, the fiber pulling experiments reveal that fibers can be pulled from the glass composition over the temperature range of about 270 to 370 °C. From 270 to 305 °C, the melt was very viscous and thick fibers were pulled. At temperatures above 305 to about 350 °C continuous fine fibers were pulled. Over the short temperature range of 350 to 370 °C one could initiate fiber pulling, but the fibers tended to break.

Short thermal fluctuations to the 420 to 470 °C temperature range caused the melt to crystallize almost instantaneously and completely. Very approximate indications of the liquidus and solidus temperatures are 540 and 640 °C, respectively. Significant volatilization was observed above 700 °C.

The above observations provide the general requirements for an acoustic levitation furnace. If one wants to melt the high purity fluoride glass precursor to make a glass preform from which fibers are pulled by conventional techniques, then one must be able to levitate at temperatures above 650 °C. On the other hand, if one chooses to pull fibers from a glass preform, one must levitate at temperatures around 300 °C. The former is well beyond the design capabilities of the terrestrial techniques. The latter is somewhat more likely to be attained.

ENGINEERING PARAMETERS OF THE ACOUSTIC LEVITATOR

In order to characterize the operation of the acoustic levitation furnace, a number of engineering parameters were measured. Figure 6 shows the actual current and voltage readings as a function of the power knob setting while Figure 7 illustrates

the actual electrical power delivered to the piezoelectric sound source as a function of the power knob setting on the front panel for both frequency "locked in" and "not locked in" conditions. The power level is roughly linear with the knob setting. Although the frequency is automatically controlled, one can overrride the automatic frequency control (AFC) by adjusting the frequency control setting. Figure 8 shows the actual signal frequency as a function of the frequency control dial setting. Over the frequency range of 22 to 25 kHz, the AFC locks in the frequency and tends to hold the frequency so as to match the oscillation frequency of the acoustic driver. Since there is some possible manual adjustment of the frequency in this range, the AFC is not entirely successful. Throughout the work the frequency was manually adjusted in order to maximize the power read on the power meter.

The levitation force was measured with a Mettler balance positioned over the furnace. A spherical sample of glass or steel was suspended by a string or wire from the balance so that it hung in the furnace below the reflector. The net force on the suspended sample was calculated by subtracting the weight of the sample, the suspending string (and wire), and the tape used to attach the assembly to the balance pan from the measured value. This net force represents a push or pull force on the sample within the acoustic fields. Plots of the net force versus position within the furnace were used to map out the acoustic fields. Figure 9 is a plot of this net force versus distance from the reflector into the furnace for a glass sphere at room temperature. For the first 1.5 in. from the reflector, the nodes are fairly regular. As one proceeds closer to the acoustic driver, the nodes become less regular and distorted by complicated harmonics. Since fiber pulling will require levitation close to the reflector, subsequent measurements were only made for the first 0.5 in. from the reflector.

Figure 10 illustrates the net force on a glass sphere at room temperature as a function of distance into the furnace for three different power settings. At 12 and 33 watts, one can see the roughly sinusoidal waveform indicating the high and low pressure acoustic nodes. At higher powers, 68 watts, the sample was levitated so well that the sample floated around in the furnace, and measured displacement does not correspond with actual distance from the reflector. Also one could not lower the sample past the first node to make power measurements. These data correspond to a reflector to acoustic driver distance that provides the most stable and maximum levitation force.

The effects of relative reflector to acoustic driver distance are represented in Figure 11. In order to overcome the levitation forces and to collect data at elevated temperature studies, heavier steel spheres were used. As the relative reflector to driver distance is varied (0.1 in. increments), the waveform of the absolute acoustic force varies greatly. This illustrates how precisely the reflector to driver distance must be adjusted in order to achieve maximum levitation force at a given acoustic node.

Temperature variations in the furnace also complicate the acoustic levitation node positions. This is shown in Figure 12 for three elevated temperatures (100, 200, and 300 °C). Each of these curves are at the "best" reflector to driver distance and frequency for the given temperature.

ACOUSTIC LEVITATOR DESIGN MODIFICATIONS

With the acoustic furnace design as received from the contractor, it was not practical to levitate the dense fluoride glass at the elevated temperatures required

for fiber pulling. Utilizing techniques previously developed by MSFC personnel, the efficiency of the levitation was greatly increased. The modification consisted of a shaped reflector which concentrated the reflected acoustic energy, thereby increasing the levitation force at the node [12]. The modification is illustrated in Figures 13 and 14. A spare Ti alloy acoustic driver was attached to the existing driver. A number of the calibration procedures were repeated for this arrangement. The electrical power versus panel power knob setting was slightly different as shown in Figure 15.

The shaped driver made it much easier to levitate samples in the furnace. The electrical power to the piezoelectric sound source required for levitation of samples was also much lower than with the flat reflector. Conditions for levitation of a number of samples in unit gravity at room temperature are shown in Table 3. The power to levitate listed in the table is the threshold value at which a levitated sample dropped out of the furnace. One could levitate silicate glass, aluminum, and the fluoride glass at the lowest power setting without their falling out of levitation. One could even levitate the densest material available, platinum with a density of 21.4 g/cm^3. Levitation at higher temperatures was also much easier as is shown in Table 4. At a given temperature, the electrical power to levitate the samples scales roughly with the density of the material.

One limitation of acoustic levitation at elevated temperatures is the fatigue life of the acoustic driver material. This is why the Ti-6Al-4V alloy was used. Even with this material one is severely limited by temperature of the furnace and the power to the driver. During the course of the experimentation, a couple of drivers were driven past the fatigue limit of the material. In order to have useful driver lifetimes the power limit must be limited. The use of the shaped reflector increases the levitation force at a given power level and makes levitation of the dense fluoride glass at elevated temperatures possible. As one can see, both steel and fluoride glass could be levitated at 300 °C within a reasonable power level.

SUMMARY AND CONCLUSIONS

An acoustic levitation furnace system has been developed, tested, improved, and demonstrated to be possible of levitating small samples of relatively dense materials to temperatures of at least 300 °C in unit gravity at power levels sufficiently small to have useful driver lifetimes. Since this is within the fiber pulling temperature range of the potential ultra-low-loss fluoride glasses, it is possible to pull fibers from containerless conditions.

Extrapolating the results to low gravity, it should also be possible to containerlessly position the fluoride glass in low gravity (10^{-3} g) and melt the fluoride-type glasses in the region of 650 °C. The complex nature of the acoustic interference levitation process makes it improbable to predict the optimum conditions. The effect of variations in the relationships between reflector to driver position, temperature, and sample shape makes it necessary to perform empirical experiments to characterize the acoustic environment. Any low-gravity experiments would, therefore, be optimized by the close interaction between an experimenter and the hardware. This could be achieved in the proposed Space Station environment.

This work is continuing under the support of the Spacelab Payload Projects Office at MSFC and the Commercial Materials Processing Office at NASA Headquarters.

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TABLE 1. CURRENT STATUS WITH EXPERIMENTAL OPTICAL COMMUNICATION FIBERS, ADAPTED FROM TEBO [4]

Material	Theoretical Loss Limit (dB/km)	Loss Achieved (dB/km)	Wavelength (μm)
Fused silica	0.2	0.2	1.5
Germanium oxide	0.1	5.0	2.0
Heavy metal oxide	0.02	-	3.0
Chalcogenide	0.01	60	4.0
Fluoride	0.001	10	4.6

TABLE 2. SELECTED PROPERTIES OF THE ZBLAL FLUORIDE GLASS, AFTER TRAN ET AL. [11]

	ZrF_4	BaF_2	LaF_3	AlF_3	LiF
Composition	53%	19%	5%	5%	20%
Properties	Density	T_g	T_x	ΔT_g	
	4.58 g/cm^3	275 °C	357 °C	82 °C	

TABLE 3. CONDITIONS FOR LEVITATION OF VARIOUS MATERIALS IN UNIT GRAVITY AT ROOM TEMPERATURE WITH A SHAPED REFLECTOR

Material	Material Density (g/cm^3)	Sample Weight (g)	Node 1		Node 2		Note
			Knob Setting	Electrical Power (W)	Knob Setting	Electrical Power (W)	
Silicate Glass	2.5	0.039	0	<12	0	<12	sphere
Al	2.7	0.031	0	<12	0	<12	disc
Fluoride Glass	5.4	0.050	10	20	0	<12	shard
Steel	7.8	0.054	10	20	1	13	sphere
Pb	12.0	0.206	38	42	14	24	sphere
Pt	21.4	0.118	not levitated		45	49	wire folded

TABLE 4. ELECTRICAL POWER (WATTS) FOR LEVITATION OF VARIOUS MATERIALS IN UNIT GRAVITY IN ELEVATED TEMPERATURES WITH A SHAPED REFLECTOR

Material	150 °C	200 °C	250 °C	300 °C	350 °C	400 °C	450 °C
Silicate Glass	13	21	25	24	27	34	53
Steel Sphere	37	-	58	57	-	-	-
Fluoride Sphere	-	-	62				
Lead Sphere	58	No	No				

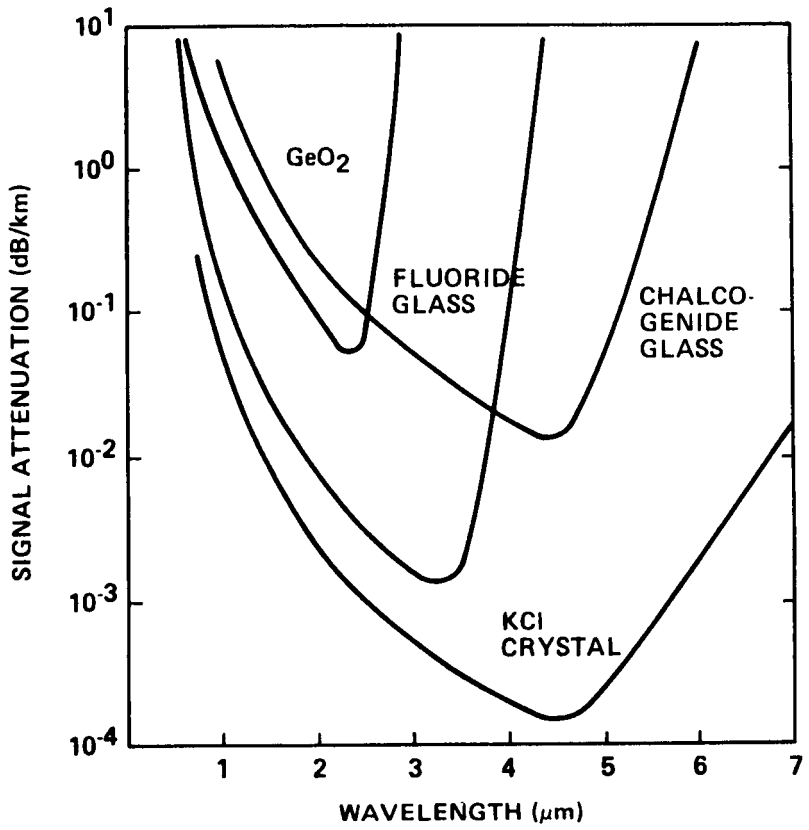

Figure 1. Theoretical spectral loss for heavy metal oxide (GeO_2), fluoride glass (ZrF_4-BaF_2-GdF_3), and crystalline KCl. Adapted from: Shibata et al. [1], Olshansky and Scherer [2], and Harrington [3].

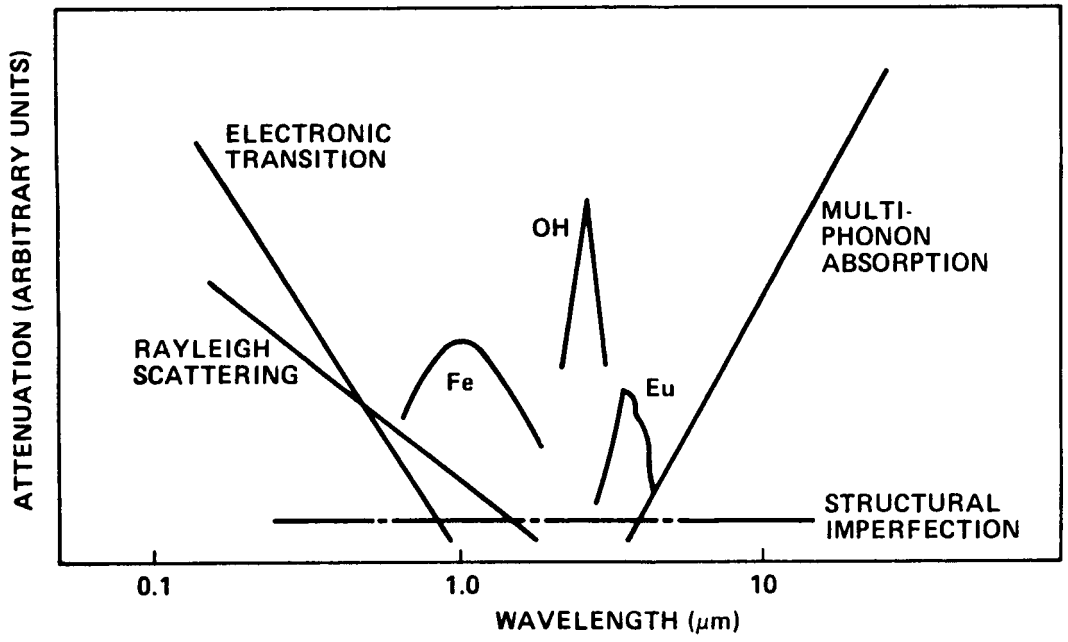

Figure 2. Schematic representation of the attenuation mechanisms in fluoride optical materials.

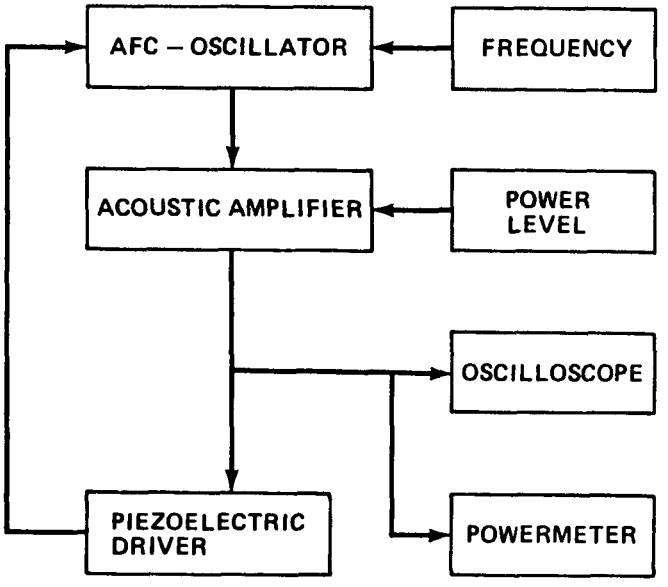

Figure 3. Schematic of the electronics subassembly of the Acoustic Levitation Furnace.

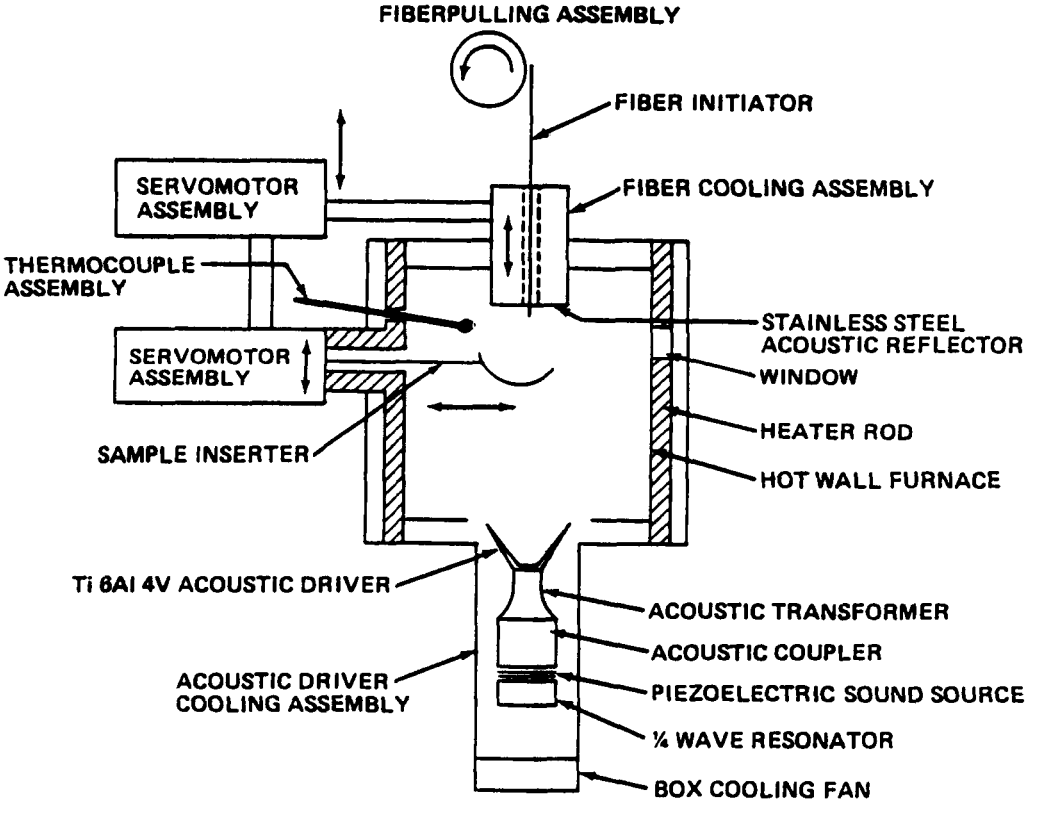

Figure 4. Diagram of the physical layout of the Acoustic Levitation Furnace.

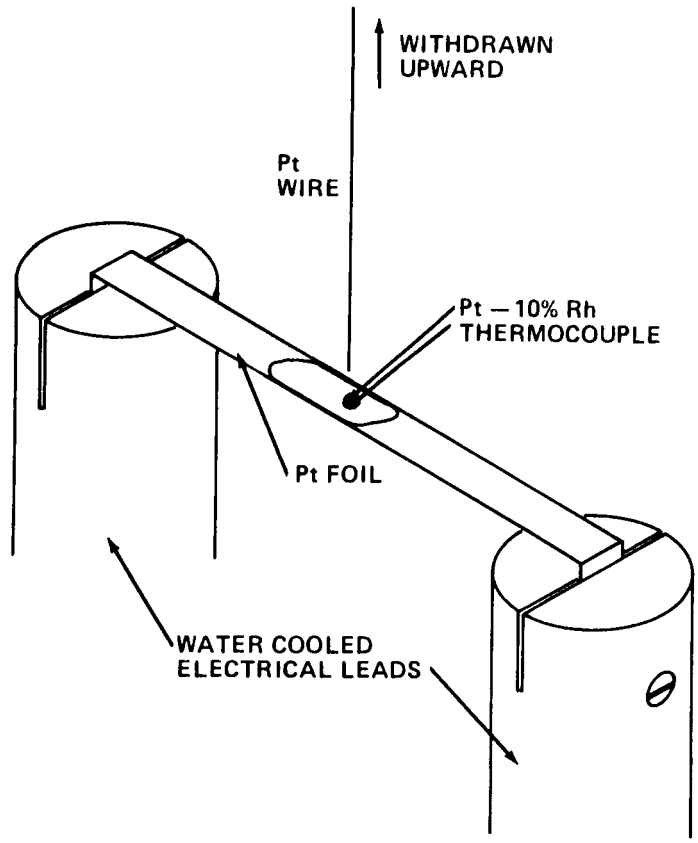

Figure 5. Platinum strip heater used for trial fiber pulling experiments of the ZBLAL type fluoride glass, F-1225.

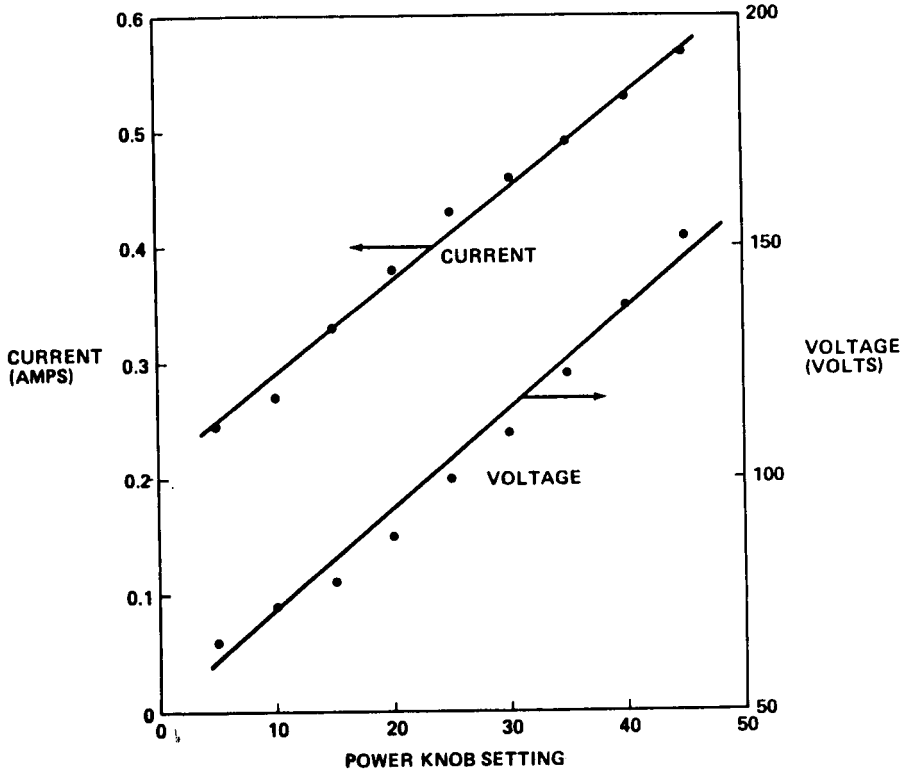

Figure 6. Current through and voltage across the piezoelectric sound source versus power knob setting.

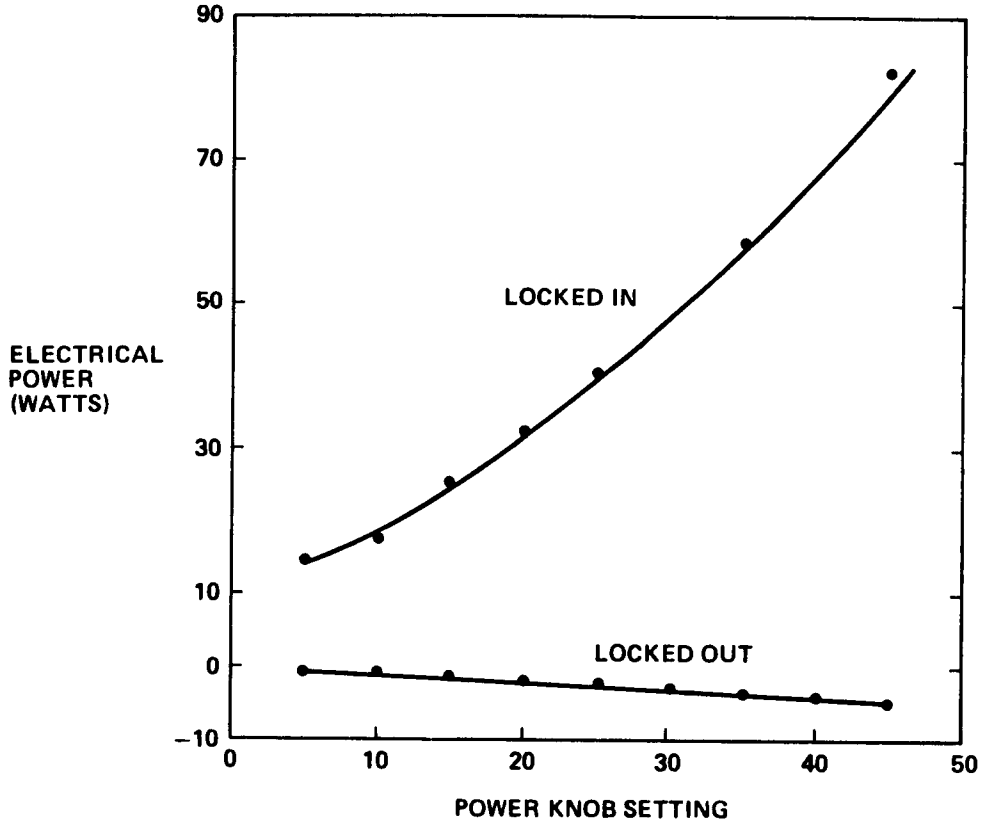

Figure 7. Electrical power versus power knob setting.

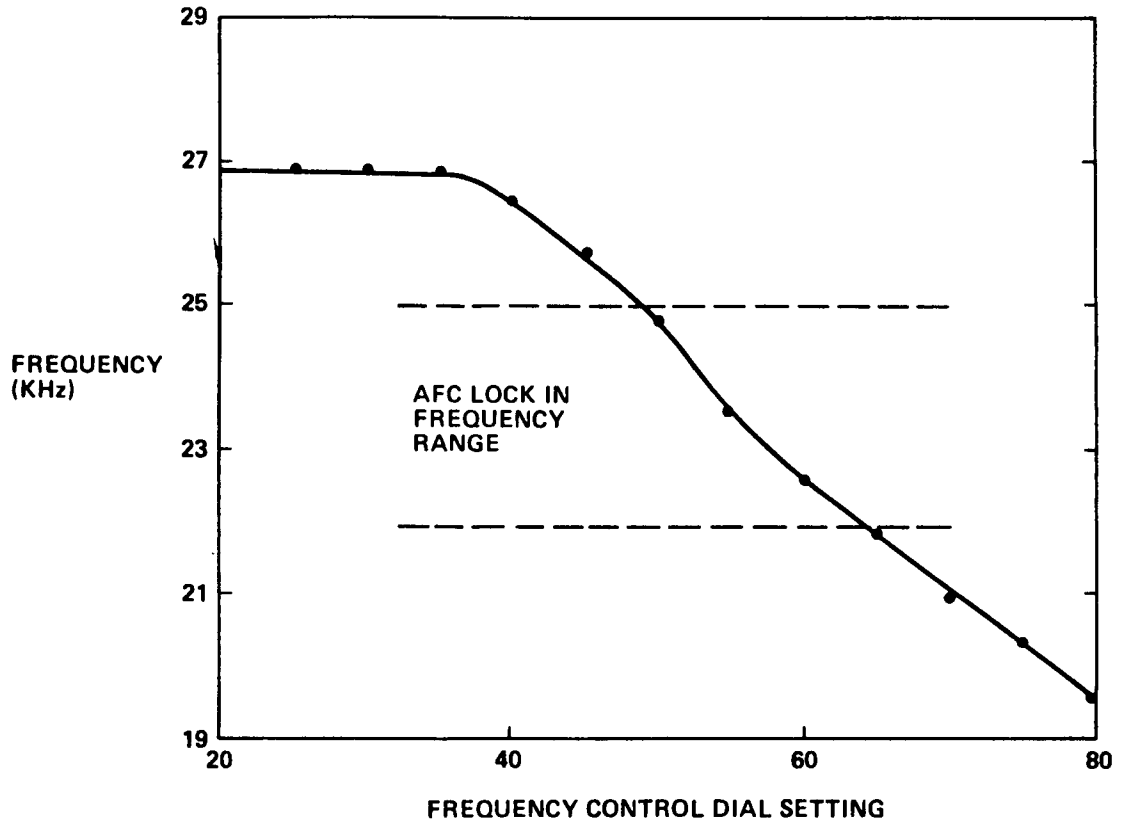

Figure 8. Acoustic signal frequency versus frequency control dial setting.

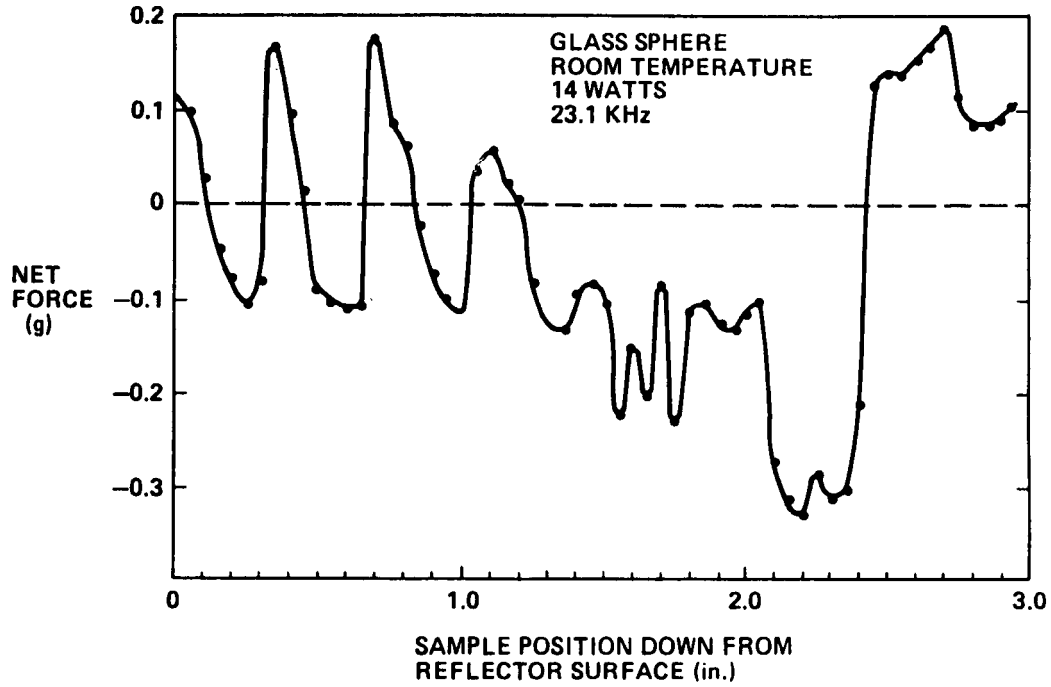

Figure 9. Plot of the net force on a glass sphere along the axis of the acoustic levitator from the reflector surface to the driver surface.

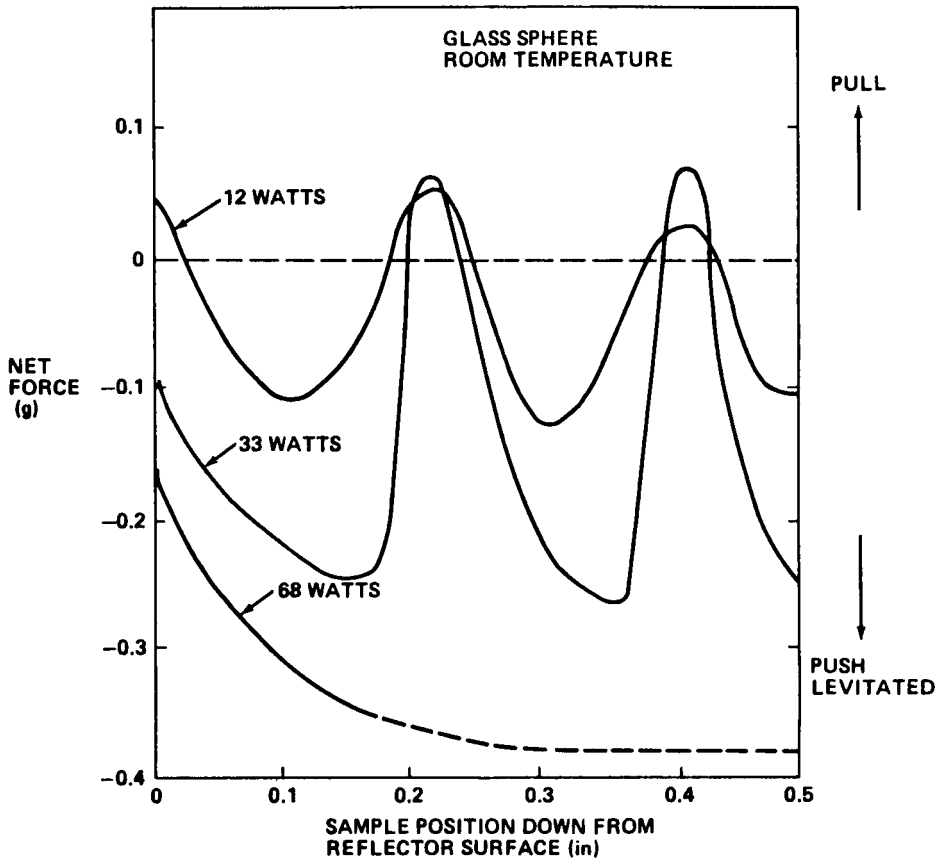

Figure 10. Plot of the net force on a glass sample versus position down from the reflector surface for three different power levels.

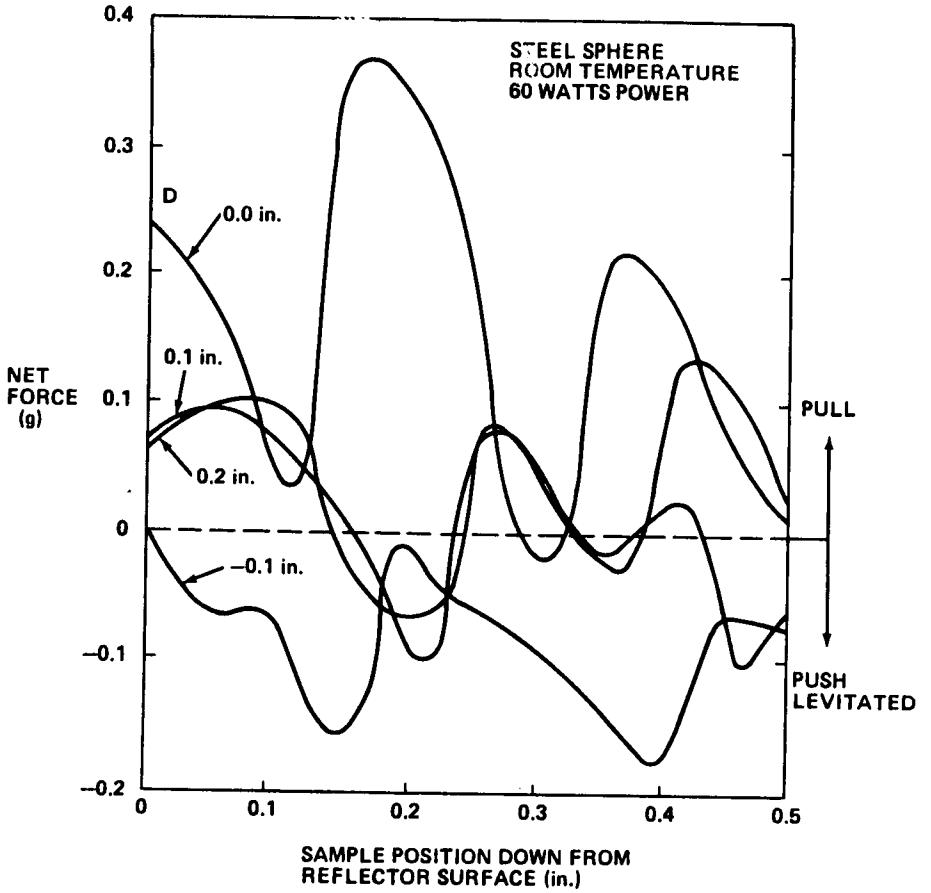

Figure 11. Plot of the net force on a steel sample versus position down from the reflector surface for four different reflector positions with respect to the driver surface.

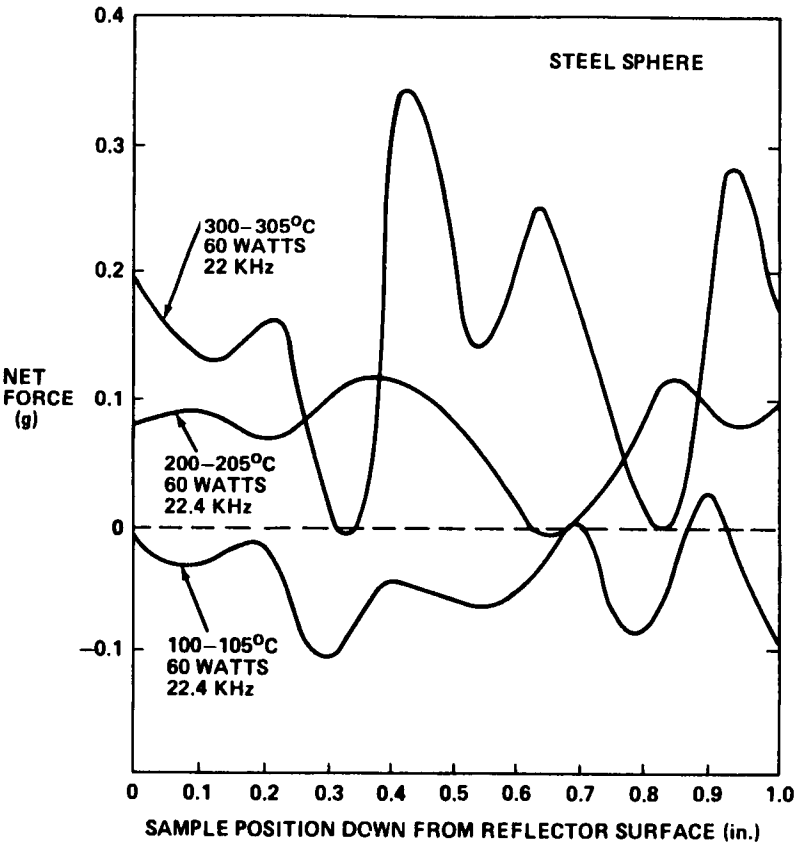

Figure 12. Plot of the net force on a steel sample versus position down from the reflector surface for three different temperatures.

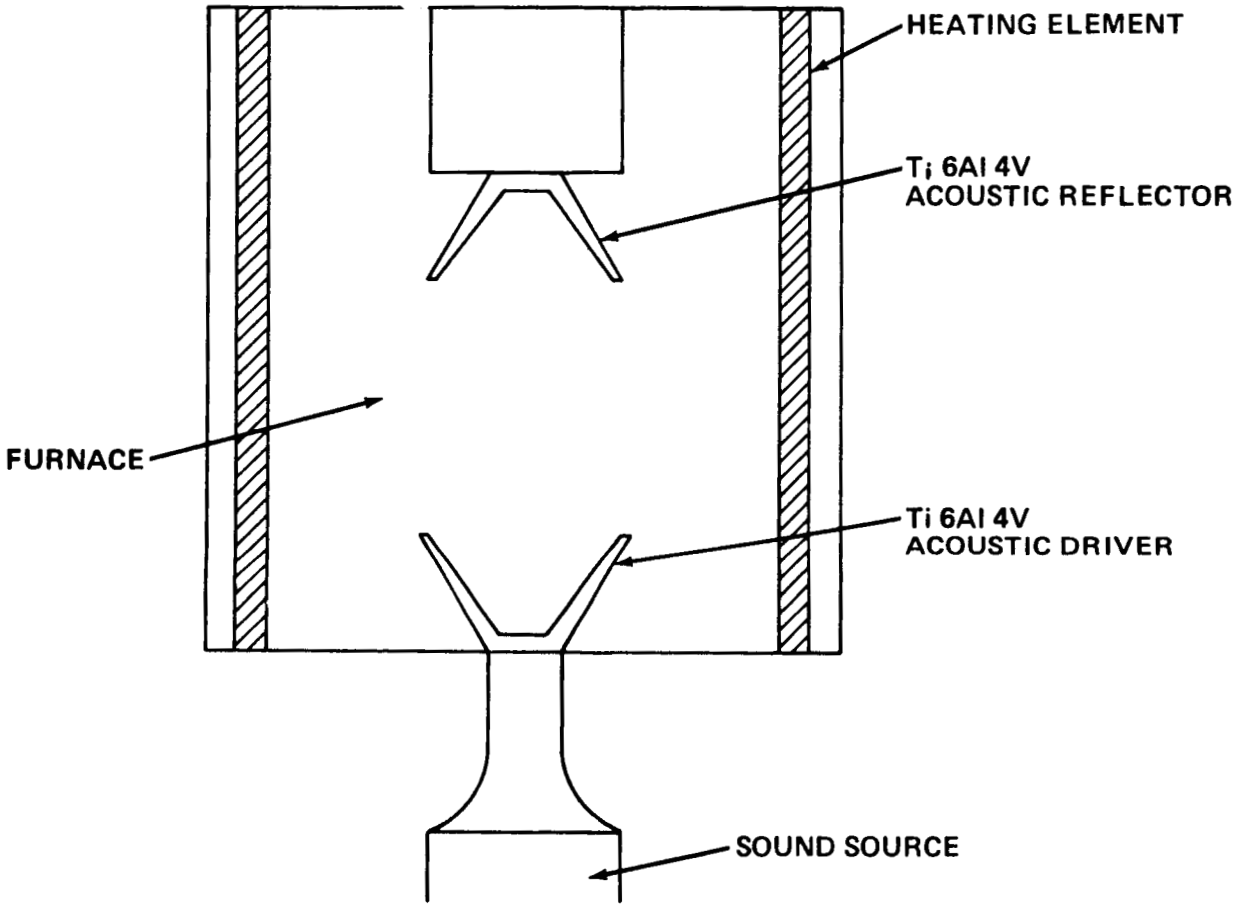

Figure 13. Diagram of the physical layout of the Acoustic Levitation Furnace with the modified shaped reflector.

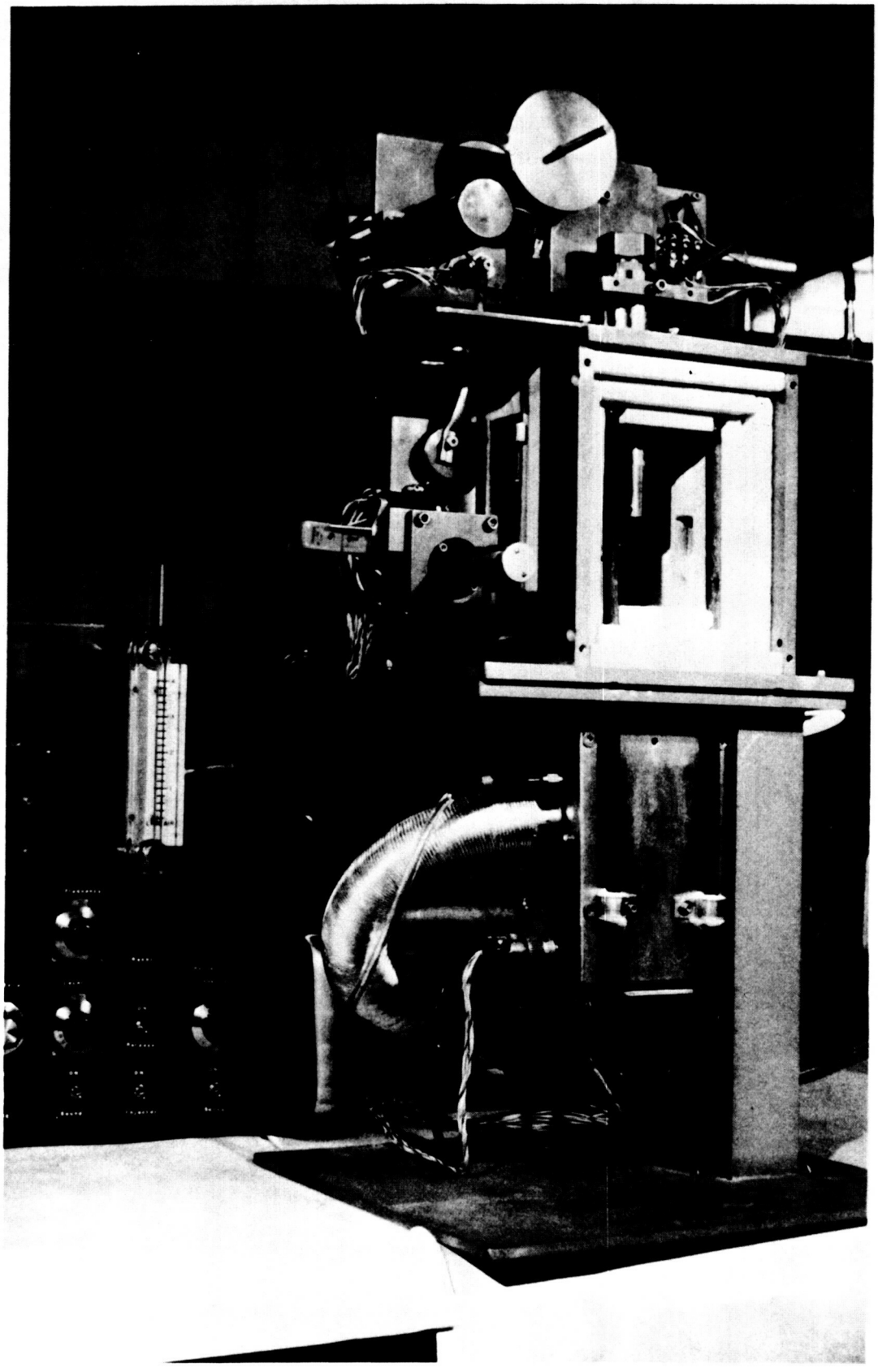

Figure 14. Photograph of the Acoustic Levitation Furnace.

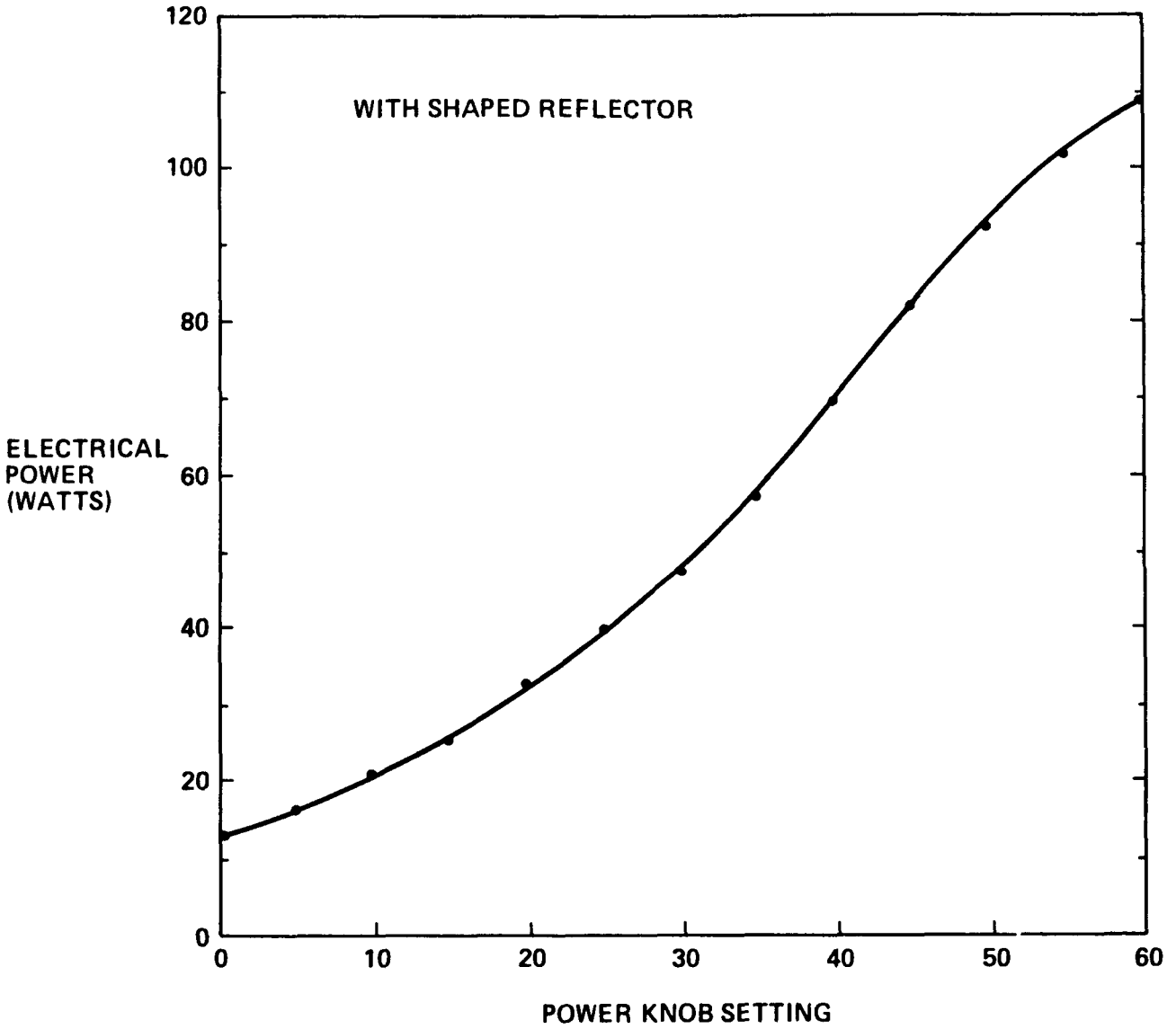

Figure 15. Electrical power versus power knob setting with the shaped reflector.

APPROVAL

CONTAINERLESS GLASS FIBER PROCESSING – MSFC CENTER DIRECTOR'S DISCRETIONARY FUND FINAL REPORT

By E. C. Ethridge and R. J. Naumann

The information in this report has been reviewed for technical content. Review of any information concerning Department of Defense or nuclear energy activities or programs has been made by the MSFC Security Classification Officer. This report, in its entirety, has been determined to be unclassified.

A. J. DESSLER
Director, Space Science Laboratory

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